PATENT SPECIFICATION

(11) 1307941

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DRAWINGS ATTACHED

(19)

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(31) Convention Application No. 10078 (32) Filed 13 Feb. 1969
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(31) Convention Application No. 101275 (32) Filed 18 Dec. 1969
(31) Convention Application No. 101276 (32) Filed 18 Dec. 1969 in
(33) Japan (JA)
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(54) A METHOD AND AN APPARATUS FOR MANUFACTURING FINE POWDERS OF METAL OR ALLOY

(71) We, SHINKU YAKIN KABUSHIKI KAISHA, a Company organised and existing under the Laws of Japan, of 300 Axa Santan-cho, Hakusan-cho, Midori-ku, Yokohama-shi, Kanagawa-ken, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us and the method by which it is to be performed to be particularly described in and by the following statement:—

in a glass bell jar forming an airtight housing. Metal or alloy of about 30 mg which is the raw material for the fine powder to be manufactured is put on the coil of the heater. An outlet opening is connected to an exhaust system, and the connection between the bell part and the base part of the bell jar is kept airtight by a gasket.

On manufacturing fine powders by means of the abovementioned apparatus, the bell

PATENTS ACT 1949

SPECIFICATION NO 1307941

Reference has been directed, in pursuance of Section 9, subsection (1) of the Patents Act 1949, to Specification No 1103442.

THE PATENT OFFICE
12 June 1973

R 69659/3

great demand for these extremely fine powders of metal and alloy in order to use them at catalysts in chemical industries, and a magnetic powder for a tape recorder. In the case of the tape of the tape recorder, if powder size of magnetic powder is small, it becomes possible to record for a long period of time under a slow speed of the tape, and for such uses the makers of tapes demand greatly such extremely fine powders. However, it is difficult to manufacture easily and in large quantities such fine powders of metal or alloy which are pure and have good qualities.

A conventional method of manufacturing such fine powders uses an apparatus which is provided with a resistance heater of tungsten in a bell jar, and in this method, a heater wound in a shape of conical coil with a tungsten wire of 0.8 mm diameter is arranged

a brush. Thus fine powders of about 10 mg. will be obtained in one operation. The size of powders varies according to a kind and a pressure of gas introduced, the state of heating, the dimension of the bell jar, and the distance between a material by said heater and the inner surface of the bell jar.

For instance, in the case of manufacturing of fine powders of iron, the following results have been obtained:

Kind of gas	Pressure in Torr	Powder size in Å
Ar	3	200
Ar	10	500
He	30	200
He	500	500

A distribution of powder size obtained by using argon as an introduced gas is better

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This invention relates to a method and an apparatus for manufacturing fine powders of metal or alloy, which are for instance under one μm in size, by heating and evaporating and/or sublimating metal or alloy.

It is well known that extremely fine powders of certain metals or alloys, the powder size of which is, for instance, under 1000 Å have superior qualities when they are used for solid materials of a magnetic element, a super conductor, a chemical catalyst, an absorber of light or electromagnetic waves, a raw material in power-metallurgy, and a semi-conductor. At present there is a great demand for these extremely fine powders of metal and alloy in order to use them at catalysts in chemical industries, and a magnetic powder for a tape recorder. In the case of the tape of the tape recorder, if powder size of magnetic powder is small, it becomes possible to record for a long period of time under a slow speed of the tape, and for such uses the makers of tapes demand greatly such extremely fine powders. However, it is difficult to manufacture easily and in large quantities such fine powders of metal or alloy which are pure and have good qualities.

A conventional method of manufacturing such fine powders uses an apparatus which is provided with a resistance heater of tungsten in a bell jar, and in this method, a heater wound in a shape of conical coil with a tungsten wire of 0.8 mm diameter is arranged in a glass bell jar forming an airtight housing. Metal or alloy of about 30 mg which is the raw material for the fine powder to be manufactured is put on the coil of the heater. An outlet opening is connected to an exhaust system, and the connection between the bell part and the base part of the bell jar is kept airtight by a gasket.

On manufacturing fine powders by means of the abovementioned apparatus, the bell jar is firstly exhausted to a vacuum of the order of 10^{-5} Torr. Then inert gas, purity of which is about 99.99% is introduced into the bell jar in such a manner that a pressure in said bell jar becomes 0.1—30 Torr for argon, 1—760 Torr for helium, and 0.1—10 Torr for xenon, respectively. Then by supplying an electric power of 100 Watt, said heater is suddenly heated. At this time said metal or alloy disperses in all directions in the bell jar in the state of vapour, is cooled on the inner surface of the bell jar, and adheres to it in form of powder. Hereupon, the bell jar is removed from its base, and the fine powder adhering to it is collected with a brush. Thus fine powders of about 10 mg. will be obtained in one operation. The size of powders varies according to a kind and a pressure of gas introduced, the state of heating, the dimension of the bell jar, and the distance between a material by said heater and the inner surface of the bell jar.

For instance, in the case of manufacturing of fine powders of iron, the following results have been obtained:

Kind of gas	Pressure in Torr	Powder size in Å
Ar	3	200
Ar	10	500
He	30	200
He	500	500

A distribution of powder size obtained by using argon as an introduced gas is better

than that obtained by using helium. In the case of helium is used and its pressure is kept below 5—6 Torr, and argon is used and its pressure is kept below 0.1 Torr, smoke is not produced, and the same phenomenon as vacuum evaporation will be presented.

On the other hand, in the case argon is used and its pressure is kept over 30 Torr, smoke will not be produced unless the materials are considerably heated. According to this manufacturing method, an output of fine powders by one operation is only 10 mg, and the wire of the heater is quickly vapourised, and for these reasons, the above said method of manufacturing is only carried out on experimental scales.

In this method there are faults in that a material of resistance wire itself is apt to evaporate and to mix with the products, and an output of fine powders are a small quantity.

Further, it has been proposed that in order to manufacture fine powders of metal or alloy, arc discharge is utilised with the anode of a material itself to be evaporated and/or sublimated, but this method is only concerned with producing powder of carbon-black or silicon carbide, or with manufacturing fine powders of metal oxide by heating and evaporating and/or sublimating a suitable metal in an ordinary atmosphere, or with manufacturing fine powders of nitride in atmosphere of nitrogen.

In one view, it is desirable that arc discharge is effected in atmosphere of non-active gases in order to manufacture the pure fine powders of metal or alloy, but as variations in the pressure of the gas and the shape of the electrode effect the discharge, in fact it is very difficult to obtain a stable condition of operation.

Furthermore, in order to increase the output of fine powders in atmosphere of gas, for Ar or N_2, it is necessary to drop down the voltage for arc discharge. However, when the voltage drops down, arc discharge expands over wide area, and therefore high temperature cannot be obtained. For these reasons, it has become clear that the method of manufacturing fine powders by arc discharge is after all unsuitable to obtain a great quantity of fine powders of metal or alloy.

The present invention has for its object to eliminate the above-mentioned faults and to provide a method and an apparatus for manufacturing pure fine powders of metal or alloy in great quantities.

According to this invention there is provided a method of manufacturing fine powders of metal or alloy comprising heating and evaporating and/or sublimating a metal or alloy in a chamber, introducing a gas flow into the chamber to one side of the surface of the evaporating and/or sublimating metal or alloy, conveying the evaporated and/or sublimated metal or alloy by means of the gas flow to a cooling means having a surface arranged on the other side of said surface of the evaporating and/or sublimating metal or alloy on which cooling surface the evaporated and/or sublimated metal or alloy cools and solidifies into a powder.

The present invention also includes an apparatus for manufacturing fine powders of metal or alloy and for carrying out the method described above, comprising an airtight container whose internal pressure can be varied, a crucible within the container for holding a metal or alloy, gas flow means to one side of the crucible for producing a gas flow which, in use, conveys evaporated and/or sublimated metal or alloy from the crucible and cooling means including a surface arranged on the other side of the crucible on which in use, evaporated and/or sublimated metal or alloy conveyed by the gas flow is cooled and solidified into a powder.

The method and apparatus according to the present invention will now be described in detail by way of example of some embodiments thereof with reference to the following drawings, in which:

Fig. 1 is a diagrammatic illustration of a first embodiment of the present invention;

Fig. 2 is a diagrammatic illustration of a second embodiment of it;

Figure 3 is a diagrammatic illustration of a third embodiment of it;

Figs. 4, 5 and 6 are diagrammatic illustrations of other embodiments of the present inventions;

Fig. 7 is a diagram relating to quantities of gas used to produce plasma jet, and illustrating a relation between a pressure of gas on a container and a quantity of fine powders produced under said pressure, and,

Fig. 8 is a diagram illustrating variation of powder size of fine powders of iron according to variation of the pressure of gas, when mixed gas of helium and hydrogen of 15% is used for producing plasma jet.

Referring now to the drawings, Fig. 1 represents diagrammatically a first embodiment of the present invention, and shows a container 1 which consists of generally cylindrical barrel 2, a front cover 3 and a rear cover 4 which are detachably connected to the ends of said barrel 2. On a lower part of said barrel 2 and near the front cover 3, there is provided a crucible 5 which is of a cooled type and on which metal or alloy 6 to be evaporated and/or sublimated are placed. On said front cover 3 is fixed a plasma gun 7 the axis of which is directed towards the metal or alloy 6 from obliquely above to apply the plasma jet 8 on it in order to evaporate and/or sublimate it. The nozzle 9 of said gun 7 opens to the inside of

the container 1. A plasma producing means is a well known technique, but in short, inert gas for producing plasma jet 8, for instance Ar, He or N_2 is introduced into the plasma gun 7 from a cylinder 10, and the quantity of flow of gas is measured with a flow meter 11 and a pressure gauge 12, and control of the quantity of flow is carried out by a valve 13. The plasma gun 7 and the crucible 5 as shown in Fig. 1 are connected to a source of direct current being able to be applied with alternating current, for instance 200 volts and when voltage is applied to the plasma gun 7 by the electric source 14, gas in the gun 7 is ionized, blown out in a form of plasma jet 8 from the nozzle 9 on to said metal or alloy 6 which being maintained in positive potential, and bombard it. In Fig. 1 numeral 15 designates an electric current controller and 16 is a starter.

The interior of the container 1 is exhausted with an exhaust pump 17, such as a rotary pump, to the pressure below the atmospheric pressure (for instance below 10^{-3} Torr), and its pressure is measured with a Geisler tube 18.

A cooling means 19 provided near the crucible 5 and is of conical shape with openings 20 and 21 on both ends, a front opening 20 being opened against the flow of evaporated and/or sublimated metal or alloy and rear opening 21 forming an insertion hole for a scraping bar 22 carrying brushes 23 adapted to scrape out fine powders adhered to the inner surface of the said cooling means. There is provided a hopper 24 which serves to receive fine powders scraped out by brushes 23, and to drop them down into an airtight vessel 25. This airtight vessel 25 prevents fine powders produced from contacting with open air, and which is particularly necessary in the case of obtaining fine powders for experimental and/or scientific research. In Fig. 1, numeral 26 represents a gas conduit connecting the cylinder 10 to the plasma gun 77, numerals 27, 28 represent respectively cooling water conduits for the said plasma gun 7, and 29, 30 represent respectively cooling water conduits for crucible 5 and said cooling means 19.

Now, the method for manufacturing fine powders according to the present invention using the abovementioned apparatus will be described.

Firstly, the container 1 is exhausted to the predetermined pressure (below 10^{-3} Torr), and then plasma producing gas are introduced into the container through the plasma gun 7 from the cylinder 10 so as to raise the pressure in the container to the pressure suitable to fine powders of metal or alloy of powder size intended to produce. In this embodiment of the present invention a mixture of helium and hydrogen is used. If the plasma gun 7 is applied with 700 volts, plasma is produced in the plasma gun 7, and it is projected from the nozzle 9 into the container 1 in the form of plasma jet or plasma gas flow 8, and applied obliquely to the metal or alloy 6 on the crucible 5, and heat and evaporate and/or sublimate them.

In this case, plasma jet or gas flow 8 being applied obliquely to the surface of metal or alloy is reflected with the angle of reflection which angle is determined according to its angle of incidence. The evaporated an/or sublimated materials are carried in the reflected plasma jet or gas flow 8 as fine powders and progress rearwards in said cooling means 19. When they are cooled in said means, they solidify and form fine grains, and therefore they adhere on the inner surface of the cooling means 19. Adhered and cold powders of metal or alloy are scraped out with brushes 23. The scraping bar 22 is arranged to be able to move to and fro, and up and down. Fine powders of metal or alloy which are scraped out slide down along the inclined bottom surface of said cooling means 19 and received in the airtight vessel 25 through said hopper 24.

An embodiment of the present invention illustrated in Fig. 2 is substantially similar to the first embodiment shown in Fig. 1 except following points that the crucible 5 in Fig. 1 is replaced with an induction heating crucible 31, and the plasma gun 7 in Fig. 1 is substituted for a hot gas projection device 32. The parts similar to that of the apparatus of Fig. 1 are represented with the similar reference number.

In this embodiment, similarly in the first embodiment, firstly, container 1 is exhausted through an exhausting port 33 by a vacuum exhauster, and then gas of helium or argon are introduced into the container. Prior to the starting of the apparatus alternating electric current is applied to coils 34 of the induction heating crucible 31, frequency of the electric current is applied to coils 34 of the induction heating crucible 31, frequency of the electric current being, if desired, high or low, therefore metal and alloy 6 in said crucible are heated and melted. Then the hot gas projection device 32 is operated, accordingly a flow of gas 35 which has been heated in the said device 32 by heating coils 36 and subsequently is effused from nozzles of a nozzle plate 37 of said heat gas projection device 32. Metal or alloy evaporated and/or sublimated will become powder or particles of solid, liquid or vapour, heated by flow of gas, and will be carried with them to the cooling means 19. After this, the operation will be progressed in such a manner as illustrated and referred to in Fig. 1.

According to this embodiment, heating of metal or alloy 6 and moving of metal or alloy evaporated and/or sublimated are affected with

different apparatus, so these two operations can be controlled independently. The characteristics of the gas are controlled and will be suited to the best condition of forming fine powders. Further, by using an induction heating crucible temperature can be very easily controlled. The apparatus according to this second embodiment can be easily realized in large scale. The induction heating crucible 31 illustrated in Fig. 2 is suitable to heat metal or alloy which do not react with the materials of the crucible.

Further according to arranging cooling means in the gas projecting device 32, it is possible to use cold gas. When ambient temperature gas or cold gas is used, the materials evaporated and/or sublimated are partially pre-cooled during being conveyed, and finally cooled and solidified in said cooling means 19.

Furthermore, all or a part of the gas may be reducing gas or oxidizing gas. By reducing gas, it is not only to prevent metal or alloy from oxidation, but even if said metal or alloy become to the state of vapour of oxidized metal or alloy, fine powders of pure metal or alloy are obtained because of being reduced. By oxidizing gas, fine grains of oxidized metal or alloy are obtained.

When two or more kinds of gases are used as heating gas, gases are introduced from each source of them, respectively, into the entrance of the said gas projection device 32, and mix during passing through it. Alternatively, a plurality of gas projecting devices may be arranged, and into thsse different devices different gas may be introduced respectively.

It is possible to mix other different metals or organic compounds in the flow of gas before said gas is projected from said gas projecting device 32. It may also be possible to arrange a plurality of induction heating crucibles along flow of gas projected from nozzle plate 37 and to put in each of said crucibles different metal or alloy. In this case, fine powders of compound or mixture of two or more kinds of metal or alloy can be obtained.

Now, the third embodiment of the present invention will be described with reference to Fig. 3. This embodiment is substantially similar to the second embodiment illustrated in Fig. 2, but it differs from the latter in that as a metal or alloy heating means there is arranged a hea source 38, and from which high energy emitted heating beam 39, such as electron beam, laser (light amplification by stimulated emission of radiation) or maser (microwave amplification by stimulated emission of radiation) are radiated directly to metal or alloy 6 in the crucible 5, said crucible having of course, no induction heating coils 34 such as are illustrated in Fig. 2. The construction and operation according to the said embodiment are substantially similar to those of the second embodiment except abovementioned points, so the description in detail will be omitted.

Instead of heating gas with resistance coils 36, it is possible to heat the gas by electric discharge. Fig. 4 represents diagrammatically the embodiment in this case, in which 40 is an electrode applied by positive voltage and 41 is an electrode applied by negative voltage. The electric discharge occurs between said both electrodes 40 and 41, the flow of gas supplied into the space between said both electrodes are ionized and directly heated, and they are projected from a nozzle 42. And gas containing ion or excited atoms or molecules is blown against metal or alloy 6. In the case of two or more kinds of gas being used as above, different kind of gas may be introduced through a hole 43 being provided in the electrode 41.

If the diameter of nozzle 42 is small, gas may be projected from the nozzle 42 and spread out even if the nozzle plate 37 is absent.

Fig. 5 represents further embodiment, in which is shown one pair of the gas projecting devices 37, the crucible 31 and the heat source 38, as viewed from above. In this embodiment, fine powders of two kinds of metal or alloy will be obtained on the cooling means 19.

Fig. 6 represents further embodiment of this invention in which an electron lens or lenses (or charged particle lens or lenses) is or are provided against the flow of gas. 44 is an electron lens which is arranged between the gas projecting device 37 and the heat source and crucible 31, 38. This lens 44 may be designed to be an electromagnetic lens which is adapted to focus flow of ionized gas and to impinge against metal or alloy in the heat source and crucible 31, 38, or it may be designed as shown in Fig. 6, to be a deflective electron lens which deflects the flow of gas. The reference number 45 represents also an electron lens being arranged between the heat source and crucible 31, 38 and the cooling means 19, and being adapted to deflect the flow of gas. If such deflecting lenses are used, positions of elements 19, 31 and 38 can be relatively freely selected. The electromagnetic lens 45 may be a diverging lens which diverges the flow of gas against the surface of the said cooling means.

In the first embodiment of the present invention (Fig. 1), quantity of fine powders being produced is greater, when the pressure in the container 1 is lower, the flow of plasma producing gas introduced into the plasma gun 7 is a smaller quantity, and electric current in said plasma gun 7 is greater. However, there is a lower limit in a value of the pressure in the container and the quantity of flow of gas in the plasma gun in order to obtain the

stable discharge. This limit value is variable according to the kind of gas. The results of test for 4 kinds of gas, that is: Ar, Ar plus 15%, H_2 15%, He, and He plus 15% (% is represented in Mol) are illustrated as follows:

	Ar	Ar+15% H_2	He	He+15% H_2
Pressure (Torr)	250	60	250	10
Quantity of flow (l/sec)	2	0.5	3	1

When rate of flow of the above gas is kept at lower limit value, and the pressure in the container is varied, rate of production of fine powders of Fe, as an example, is represented on Fig. 7. As shown in this diagram, rates of production are lowered in order of the case of He+15% H_2, He, Ar+15% H_2, and Ar, and rate of production of fine powders produced in atmosphere of He+H_2 is considerably different from that of fine powders produced in atmosphere of Ar. This diagram also shows that rate of production of fine powders does not change even if the pressure in container is increased in the case of atmosphere of He+15% H_2 and He, but the rate of production of fine powders products is decreased, with increase in pressure in the case of Ar+15% H_2, and Ar. And according to this diagram, it is clear that the rate of production in the case of He or Ar added with 15% H_2 is greater than that in the case of He or Ar being used independently.

In the above test, the reason of that the quantity of H_2 to be added into He (or Ar) is limited to 15% is due to the construction of plasma gun as described after. This invention is not limited to said value.

The advantage due to addition of H_2 is as follows:—

a) plasma jet becomes more stable;
b) the temperature of plasma jet becomes higher, accordingly the quantity of evaporated metal is increased, and
c) oxidation of produced fine powders can be prevented.

In the case of using the mixture of He gas and H_2 gas and in the case of using the He gas only, the rate of production of powders does not vary with changes in the pressure of gas over a range of 10—800 Torr, as shown in Fig. 7.

The quantity of H_2 required must be varied according to the construction of the plasma gun and is normally in the range of 10% to 80% by volume. The state of stability of plasma jet is effected by the construction of the plasma gun, accordingly in order to obtain the best condition of plasma jet stability it is necessary to control the quantity of addition of H_2. For this reason, in the abovementioned test, it was determined that the proper value of the quantity of addition of H_2 was 15%.

Now, the reference between the powder size of fine powders of metal and the pressure in the container will be described. If the pressure in the container is higher, the powder size of fine powders produced is greater. When (He+15% H_2) gas are used and the pressure in the container is varied, mean diameter Å of powder size of produced fine powders of Fe, as an example, is represented in Fig. 8. It is recognised that when the pressure in the container is low pressure such as 200 Torr, said mean diameter of powder size is 200 Å, and when the pressure in the container is high such as 800 Torr, said diameter becomes about 1000 Å, and the powder size varies linearly due to the pressure. This reason is that when the pressure is high, in other words, the density of gas molecule becomes great, the mean free path is short, the occasion that gas molecules combine in accordance with their collision each other before their reaching to the inner surface of the container, accordingly the powder size of products. Thus, it is possible to obtain fine powders of metal or alloy of the desired powder size on the base of the test value as illustrated in Fig. 8.

It is advantageous for cooling and solidifying metal or alloy evaporated and/or sublimated to make the incident angle of plasma jet against the evaporated material (the angle between plasma jet and the surface of metal or alloy) a small angle, since this minimizes the distance which the reflected gas jet passes before coming under the influence of the cooling means.

As abovementioned, according to the embodiment of the invention in which comprising to use the mixed gas of He and H_2 as a plasma producing gas, to heat and evaporate and/or sublimate the metal with plasma jet and to cool them, a great quantity of fine powders of metal or alloy and desired size of fine powders can be obtained.

WHAT WE CLAIM IS:—

1. A method of manufacturing fine powders of metal or alloy comprising heating and evaporating and/or sublimating a metal or alloy in a chamber, introducing a gas flow into the chamber to one side of the surface of the evaporating and/or sublimating metal or alloy, conveying the evaporated and/or sublimated metal or alloy by means of the gas flow to a cooling means having a surface arranged on the other side of said surface of the evaporating and/or sublimating metal or alloy on which cooling surface the evaporated and/or sublimated metal or alloys cools and solidifies into a powder.

2. A method according to a claim wherein the gas flow comprises a mixture of helium with 10% to 80% of hydrogen by volume, said mixture being blown in a plasma jet from a plasma gun onto the metal or alloy.

3. A method according to claim 1 or claim 2 wherein the atmosphere surrounding the metal or alloy is inert and the pressure of the atmosphere is varied to vary the size of the powdered metal or alloy produced.

4. Apparatus for manufacturing fine powder of metal or alloy and for carrying out the method of claim 1, comprising an air-tight container whose internal pressure can be varied, a crucible within the container for holding a metal or alloy, gas flow means to one side of the crucible for producing a gas flow which, in use, conveys evaporated and/or sublimated metal or alloy from the crucible and cooling means including a surface arranged on the other side of the crucible on which in use, evaporated and/or sublimated metal or alloy conveyed by the gas flow is cooled and solidified into a powder.

5. Apparatus according to claim 4 wherein the gas flow means comprise a plasma producing device which is arranged, in use, to direct a plasma jet obliquely against said metal or alloy.

6. Apparatus according to claim 4 wherein the crucible is inductively heated.

7. Apparatus according to claim 6 wherein the gas flow means includes a heating device so that, in use, the gas flow is heated.

8. Apparatus according to claim 4 wherein the crucible is cooled and wherein there is provided a heating device which, in use, emits a high energy electromagnetic or electron beam which is directed on to the metal or alloy to heat the metal or alloy.

9. Method according to claim 1 for manufacturing fine powders of metal or alloy, the method being substantially as herein particularly described with reference to any of the alternatives of the accompanying drawings.

10. Fine powders of metal or alloy when produced by a method in accordance with any of claims 1 to 3 or 8.

11. Apparatus for manufacturing fine powders of metal or alloy, the apparatus being constructed and arranged substantially as herein particularly described with reference to any one of the alternatives shown in the accompanying drawings.

BREWER & SON,
Chartered Patent Agents,
5—9 Quality Court,
Chancery Lane,
London, W.C.2.

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FIG. 1

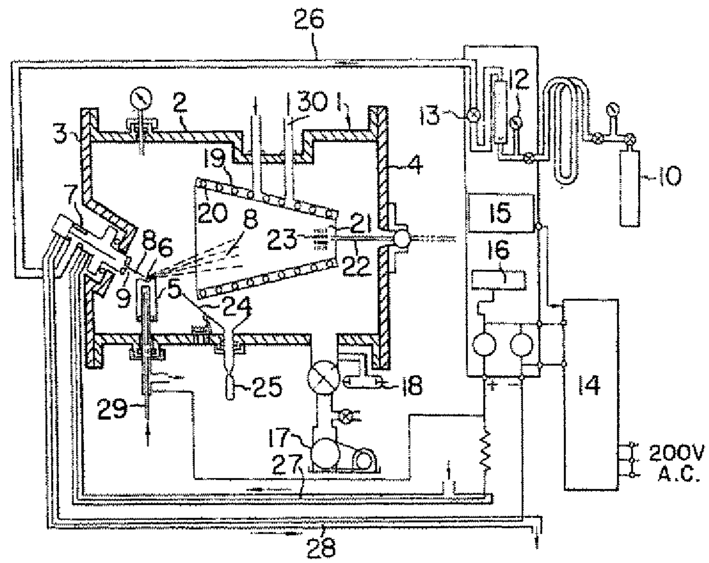
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FIG. 2

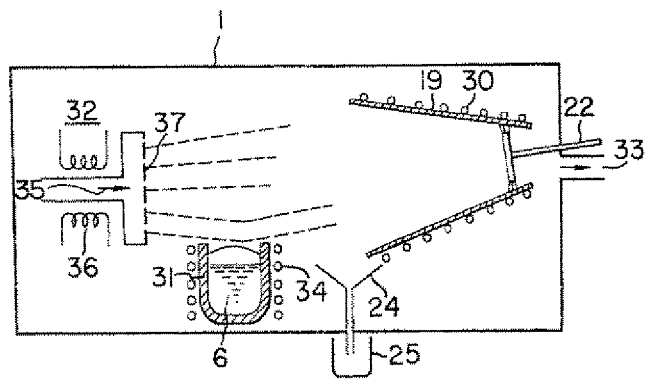
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FIG. 3

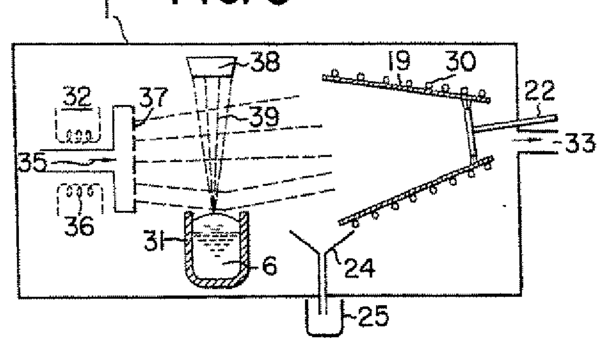


FIG. 4

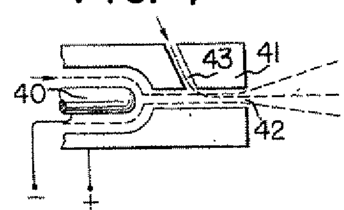


FIG. 5

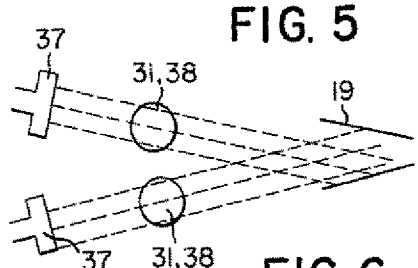


FIG. 6

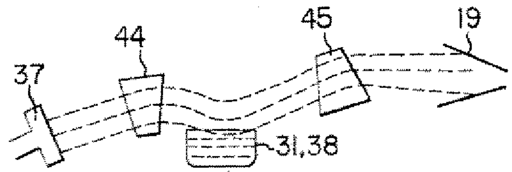


FIG. 7

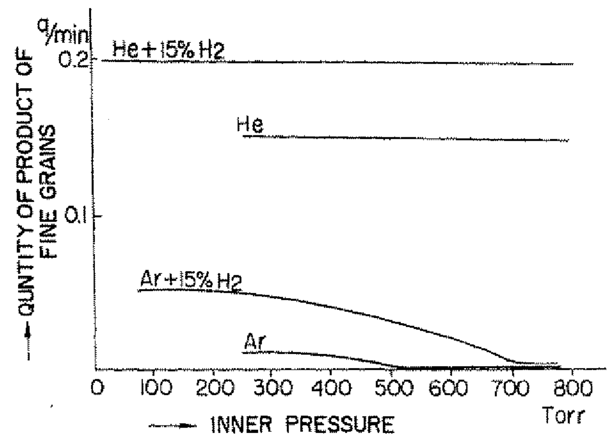
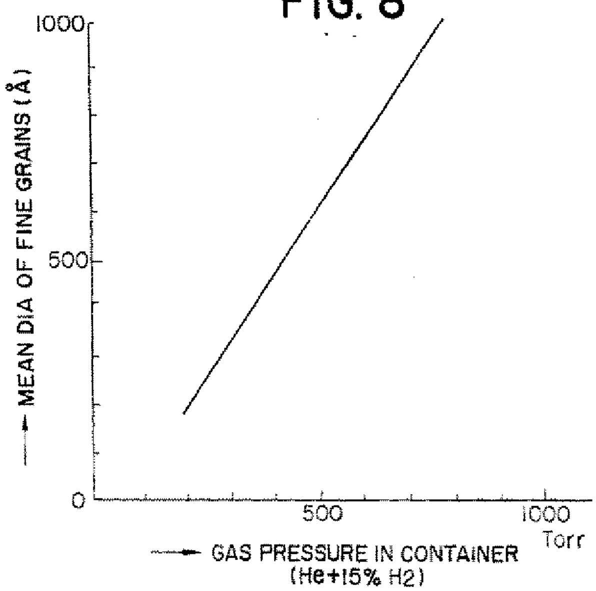

g/min
0.2
0.1
He+15% H2
He
Ar+15% H2
Ar
QUNTITY OF PRODUCT OF FINE GRAINS
0
100
200
300
400
500
600
700
800
Torr
INNER PRESSURE

FIG. 8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MEAN DIA OF FINE GRAINS (Å)
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Torr
GAS PRESSURE IN CONTAINER
(He+15% H2)